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Indian Standard
SPECIFICATION FOR PHENTHOATE, TECHNICAL

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3 *April* 1977

Indian Standard

SPECIFICATION FOR PHENTHOATE, TECHNICAL

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AMENDMENT NO. 1 JUNE 1989

TO

IS : 8293 - 1976 SPECIFICATION FOR PHENTHOATE, TECHNICAL

(*Page* 6, *Appendix* A) — Insert the following in the beginning:

A-0. GENERAL

A-0.1 Either of the two methods, namely, Gas Chromatographic Method (GLC) (*see* A-1) or Thin Layer Chromatograph Method (TLC) (*see* A-2) may be used for determination of phenthoate content. GLC method shall be the referee method in case of dispute.

A-1. GLC METHOD

A-1.1 Principle — A GLC unit fitted with a flame ionization detector is used for this determination. Using solutions containing known amounts of phenthoate standard and internal standard, the response factor, *RF*, for phenthoate and internal standard is arrived at. A solution containing a known mass of sample and internal standard is injected subsequently into the GLC unit. The percentage of phenthoate is then calculated by standard relationship.

A-1.2 Apparatus

A-1.2.1 *Gas Liquid Chromatograph* (*GLC*) — Equipped with flame ionization detector (FID). The GLC unit may be fitted with a printer-plotter *cum* integrator. The suggested operative parameters are given below but can be changed in any other equipment, provided standardization is done:

Column : 180 cm length — 3 mm ID

Glass/stainless steel packed with 3 percent SE-30 on chromosorb W-HP, 100-120 mesh.

Carrier gas : Nitrogen 34 - 40 ml per minute flow rate

Temperature : Column 220°C

Injection port 230°C

Detector 240°C

A-1.2.2 *Microlitre Syringe* — 10 µl capacity.

A-1.3 Reagent

A-1.3.1 *Methanol* — Analytical reagent or spectroscopy grade.

A-1.3.2 *Internal Standard* — Dibutyl phthalate.

A-1 3.3 *Dichloromethane* — Spectroscopy grade.

A-1.3.4 *Phenthoate Standard* — of known purity.

A-1.4 Preparation of Solutions

A-1.4.1 *Calibration Solution* — Weigh accurately to the nearest 0·1 mg, 0·180 g of internal standard and 0·250 g of phenthoate standard into a 25-ml volumetric flask, dissolve in methanol and make up to the mark. Mix well.

A-1.4.2 *Sample Solution*

A-1.4.2.1 *For phenthoate, technical* — Weigh accurately to the nearest 0·1 mg, 0·180 g of internal standard and 0·250 g of phenthoate technical into a 25-ml volumetric flask, dissolve in dichloromethane and make up the volume. Mix well.

A-1.4.2.2 *For phenthoate, EC* — Weigh accurately to the nearest 0·1 mg, 0·180 g of internal standard and 0·490 g of EC, dissolve in dichloromethane and make up the volume. Mix well.

A-1.4.3 *Estimation* — Inject 2 μl of the calibration (A-1.4.1) and the sample (A-1.4.2.1 or A-1.4.2.2) solutions in a proper sequence. Proceed with the next injection only 2-3 minutes after the emergence of the phenthoate peak. From the injections of the calibration solution, calculate the response factor *RF*.

A-1.5 Calculation — $RF = \frac{W_1 \times A_1 \times P_1}{W_2 \times A_2 \times P_2}$

$$\text{Phenthoate content, percent by mass} = \frac{A_3 \times W_1 \times P_1}{A_2 \times W_3 \times RF}$$

where

A_1 = area of phenthoate standard;

A_2 = area of internal standard;

A_3 = area of sample;

W_1 = mass, in g, of internal standard;

W_2 = mass, in g, of phenthoate standard;

W'_3 = mass, in g, of sample;

P_1 = percent purity of internal standard; and

P_2 = percent purity of phenthoate standard.

(*Page* 6, *clause* **A-0**) — Substitute '**A-2.0, A-2.0.1, A-2.1, A-2.1.1, A-2.1.2, A-2.1.3, A-2.1.4, A-2.1.5, A-2.1.6, A-2.1.7, A-2.1.8, A-2.2, A-2.2.1, A-2.2.2, A-2.2.3, A-2.2.4, A-2.2.5, A-2.2.6, A-2.2.7, A-2.2.8, A-2.2.9, A-2.3, A-2.3.1, A-2.3.2, A-2.3.3, A-2.3.4, A-2.3.5, A-2.3.6, A-2.3.7,** and **A-2.4**' *for* '**A-0, A-0.1, A-1, A-1.1, A-1.2, A-1.3, A-1.4, A-1.5, A-1.6, A-1.7, A-1.8, A-2, A-2.1, A-2.2, A-2.3, A-2.4, A-2.5, A-2.6, A-2.7, A-2.8, A-2.9, A-3, A-3.1, A-3.2, A-3.3, A-3.4, A-3.5, A-3.6, A-3.7** and **A-4**' respectively.

(AFCDC 6)

Power Printers, Phones: 23272445, 23283911

Indian Standard

SPECIFICATION FOR PHENTHOATE, TECHNICAL

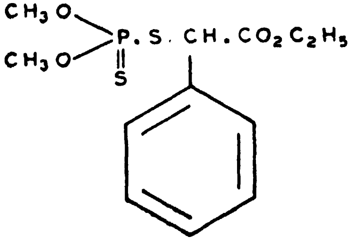
0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 31 December 1976, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Phenthoate is a non-systemic insecticide, with contact and stomach action, and is used against a broad range of insect pests of agricultural importance.

0.3 Phenthoate is the accepted common name by the International Organization for Standardization for S-α-ethoxycarbonylbenzyl 0, 0-dimethyl phosphorodithioate. The empirical and structural formulae and the molecular mass of phenthoate are as indicated below:

Empirical Formula	*Structural Formula*	*Molecular Mass*
$C_{12}H_{17}O_4PS_2$		320



0.4 In the preparation of this standard, due consideration has been given to the provisions of the Insecticides Act, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with

IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for phenthoate, technical, employed in the preparation of pesticidal formulations.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of an oily brownish-yellow liquid with an aromatic odour, free from extraneous impurities or added modifying agents.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR PHENTHOATE, TECHNICAL

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Appendix of This Standard	Cl No of IS : 6940-1973*
(1)	(2)	(3)	(4)	(5)
i)	Phenthoate content, percent by mass, *Min*	90	A	—
ii)	Material insoluble in acetone, percent by mass, *Max*	0·5	—	9
iii)	Moisture, percent by mass, *Max*	0·5	—	4.1
iv)	Acidity (as H_2SO_4), percent by mass, *Max*	1·0	—	11.3.2
v)	Specific gravity at 27°C	1·22 to 1·23	—	5

*Methods of tests for pesticides and their formulations.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in clean and dry containers made of mild steel suitably and properly lacquered from inside. The containers shall also comply with general requirements as stipulated in **2** of IS : 8190 (Part II)-1976†.

---

*Rules for rounding off numerical values (*revised*).

†Requirements for packing of pesticides: Part II Liquid pesticides.

3.2 Marking — The containers shall bear legibly and indelibly the following information in addition to the provisions of the Insecticides Act and the Rules:

a) Name of the material;
b) Name of the manufacturer;
c) Date of manufacture;
d) Batch number;
e) Phenthoate content, percent (*m*/*m*);
f) Net mass of contents; and
g) The minimum cautionary notice worded as under:

'POISONOUS. HANDLE WITH CARE. KEEP MATERIAL OUT OF REACH OF CHILDREN AND WELL AWAY FROM FOODSTUFFS, ANIMAL FEEDS, AND THEIR CONTAINERS. AVOID SKIN CONTACT. WHILE HANDLING WEAR PROTECTIVE GLOVES AND CLEAN PROTECTIVE CLOTHING. IN CASE OF POISONING CALL THE PHYSICIAN. SPECIFIC ANTIDOTES — ATROPINE AND PRALIDOXIME.'

3.2.1 The container shall also be marked with the symbol for danger of poisoning as specified in IS : 1260 (Part I)-1973*.

3.2.2 The product may also be marked with Standard Mark.

3.2.3 The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufactures or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 The method of drawing representative samples of the material shall be as prescribed in the ' Indian Standard method for sampling of pesticides and their formulations ' (*under preparation*).

NOTE — Till such time the standard under preparation is published, the matter shall be as agreed to between the concerned parties.

5. TESTS

5.1 Tests shall be carried out as prescribed in col 4 and 5 of Table 1.

---

*Pictorial markings for handling and labelling of goods : Part I Dangerous goods (*first revision*).

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

[*Table* 1, *Item* (i)]

DETERMINATION OF PHENTHOATE CONTENT

A-0. PRINCIPLE

A-0.1 The method outlined is based on the principle of separation of the active ingredient by the thin layer chromatographic technique and subsequent estimation of phosphorus in the appropriate spot as ammonium phosphomolybdate.

A-1. APPARATUS

A-1.1 Thin Layer Chromatographic Glass Plates — 20 × 20 cm.

A-1.2 Adjustable Thin-Plate Applicator

A-1.3 Chromatographic Spray Bottle

A-1.4 Chromatographic Chamber — 22 × 10 × 22 cm.

A-1.5 Oven — with a thermo-regulator.

A-1.6 Vacuum Rotating Evaporator

A-1.7 Vacuum Filtration Apparatus

A-1.8 Kjeldahl, Digestion Flask — 50 ml capacity.

A-2. REAGENTS

A-2.1 Adsorbent — silica gel G, chromatographic grade.

A-2.2 Mixed Solvent — prepared by mixing *n*-hexane and acetone in proportion of 90 and 10 parts (*v/v*) respectively.

A-2.3 Chromogenic Reagent — prepared by dissolving 0·25 g of palladium(ous) chloride in 1 ml of concentrated hydrochloric acid and 5 ml of water. The solution is then made upto 100 ml with 95 percent ethanol.

A-2.4 Benzene

A-2.5 Concentrated Sulphuric Acid — sp gr 1·84.

A-2.6 Concentrated Nitric Acid — sp gr 1·36.

A-2.7 Dilute Nitric Acid — 1 : 1.

---

*Specification for water, distilled quality (*revised*).

A-2.8 Ammonium Nitrate Solution — prepared by dissolving 20 g of ammonium nitrate in water, and then solution made up to 1 litre. Filter before use.

A-2.9 Molybdate Reagent — Dissolve 300 g of ammonium molybdate in 798 ml of boiling water. Let it cool. Add 100·2 g of ammonium sulphate and 1 002 ml of concentrated nitric acid. Keep undisturbed in dark for 72 h and dilute to 2 litre. Filter the solution before use.

A-3. PROCEDURE

A-3.1 Preparation of Chromatographic Plates — Mix 50 g of silica gel with 85-90 ml of water for three minutes. Then, by means of the applicator, coat the plate with the silica gel layer of thickness not exceeding one millimetre, and place it in the oven maintained at 110°C for 90 minutes. By means of a needle, mark a thin line widthwise on all plates at about 3 cm from the upper edge. Also remove a band of about 5 mm width silica gel from either of the lateral sides of each plate.

A-3.2 Preparation of the Sample Solution — Homogenize the sample for a few minutes, and weigh by means of an acid weighing bottle 1·0 g of the material and transfer to a 50-ml volumetric flask. Dissolve in benzene and make up to the solution to the mark by adding additional quantity of benzene.

A-3.3 Chromatographic Separation of Phenthoate — Pipette 1 ml of the benzene solution (**A-3.2**) in the form of a uniform line of about 14 cm length on the plate, at a distance of 3 cm from the bottom edge. Rinse externally the tip of the pipette with benzene using a glass capillary. After complete evaporation of the solvent, place the plate in the chromatographic chamber and allow the mixed solvent in the chamber to migrate, until it reaches the upper line of the plate. Remove the plate and let it dry in air for at least 30 minutes. Repeat this operation four times by maintaining the volume of the mixed solvent in the tank at its original level. Dry plate completely and spray with chromogenic reagent and let it stand for at least 3 hours. The separated spot characteristic of phenthoate can be identified by a bright yellow band.

A-3.4 Quantitative Estimation — Two hours after the development of the plate mark the area corresponding to phenthoate. Remove completely the silica gel outside of the considered zone by scrapping and then clean by a filter paper moistened with methanol. Then scrap off the phenthoate area, and transfer quantitatively the silica gel in a glass crucible mounted on the vacuum assembly and elute with 30-40 ml methanol, allowing the solvent to remain for a few minutes in contact with the silica gel before filtering. Carry out the vacuum filtration and collect the filtrate directly in the Kjeldahl flask. After each washing with methanol, release vacuum and stir carefully by a glass rod, the silica gel in the crucible. Evaporate the filtrate using the rotating evaporator, care being taken that the temperature at no time exceeds 30°C. After drying, add 4·5 ml of concentrated sulphuric acid followed by 1·5 ml of concentrated nitric acid. Heat gently to white fumes and cool. Add 1 ml of concentrated nitric acid and repeat digestion again to white fumes. Cool to room temperature and transfer to a 600-ml beaker. Rinse carefully the flask with water. Add 8 ml of concentrated nitric acid, dilute to 150 ml and heat beaker over steam bath. Remove from steam bath and promptly pipette 150 ml of molybdate reagent into the centre of the digest (*not down the beaker walls*). After 3 to 4 minutes, swirl gently, cover the beaker and let it stand in a dark place for not less than 12 hours.

A-3.5 Wash another glass crucible with dilute ammonium hydroxide solution, water, then with dilute warm nitric acid and again with water. Finally remove traces of water using twice 25 ml of ethanol and then 25 ml of acetone (release vacuum after each washing).

A-3.6 Suck dry, place the crucible in a vacuum desiccator containing no desiccant and evacuate to 15-30 mmHg for 30-35 minutes. Release vacuum, and weigh the crucible five minutes after it has been removed from the desiccator.

A-3.7 Filter the precipitate and wash with about 400 ml of ammonium nitrate solution. Then wash five times with 25 ml portion of ethanol and twice with 25 ml portion of acetone. Finally, continue as described for weighing empty crucible (*see* **A-3.6**).

A-4. CALCULATION

$$\text{Phenthoate content, percent by mass} = \frac{10{\cdot}34 \times P \times 100 \times 0{\cdot}016\,5}{M}$$

where

P = mass in g of ammonium phosphomolybdate, and

M = mass in g of sample applied to the chromatographic plate.

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Savitri Complex, 116 G.T. Road, GHAZIABAD 201001	286 1498
53/5 Ward No. 29, R.G. Barua Road, 5th By-lane, Apurba Sinha Path, GUWAHATI 781003	245 6508
5-8-56C, L.N. Gupta Marg, Nampally Station Road, HYDERABAD 500001	2320 1084
Prithavi Raj Road, Opposite Bharat Overseas Bank, C-Scheme, JAIPUR 302001	222 3282
11/418 B, Sarvodaya Nagar, KANPUR 208005	223 3012
Sethi Bhawan, 2nd Floor, Behind Leela Cinema, Naval Kishore Road, LUCKNOW 226001	261 8923
H. No. 15, Sector-3, PARWANOO, Distt. Solan (H.P.) 173220	235 436
Plot No A-20-21, Institutional Area, Sector 62, Goutam Budh Nagar, NOIDA 201307	240 2206
Patliputra Industrial Estate, PATNA 800013	226 2808
Plot Nos. 657-660, Market Yard, Gultkdi, PUNE 411037	2427 4804
"Sahajanand House" 3rd Floor, Bhaktinagar Circle, 80 Feet Road, RAJKOT 360002	237 8251
T.C. No. 2/275 (1 & 2), Near Food Corporation of India, Kesavadasapuram-Ulloor Road, Kesavadasapuram, THIRUVANANTHAPURAM 695004	255 7914
1st Floor, Udyog Bhavan, VUDA, Siripuram Junction, VISHAKHAPATNAM-03	271 2833

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*Sales Office is at 5 Chcwringhee Approach, P.O. Princep Street, KOLKATA 700072 2355 3243

†Sales Office (WRO) Plot No. E-9, MIDC, Rd No. 8, Behind Telephone Exchange, Andheri (East), Mumbai-400 0093 2832 9295

Power Printers, Phones: 23272445, 23283911